



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. of: Robert J. SMALL and Zhefei J. CHEN Confirmation No: 1702
Serial No: 10/057,206 Group: 1755
Filed: January 25, 2002 Examiner: M. Marcheschi
Entitled: **Compositions for Chemical Mechanical
Planarization of Noble-Metal-Featured
Substrates, Associated Methods, and
Substrates Produced by Such Methods** Atty Docket: 63254-5002-US

DECLARATION OF ROBERT J. SMALL AND ZHEFEI J. CHEN
UNDER 37 C.F.R. § 1.131

We, ROBERT J. SMALL and ZHEFEI J. CHEN declare as follows:

1. We are co-inventors named in the above-referenced patent application, which was filed on January 25, 2002 ("the Application").

2. We were employed by EKC Technology, Inc., located in Hayward, California and to whom we assigned the invention described in the Application. As part of our work for EKC, we developed and tested compositions for polishing a substrate having a noble metal material. The compositions comprised periodic acid or H_5IO_6 , which was sometimes referred to as "PIA," and an abrasive. The compositions that we developed are the subject matter of the Application and all of our work regarding the development and testing of these compositions was done in the United States of America.

3. We understand that independent claims 77 and 112 in the Application stand rejected as being obvious over U.S. Patent No. 6,589,100 ("Moeggenborg 100") in view of U.S. Patent No. 6,461,227 ("Fang") and U.S. Patent Application Publication No. 2002/0076932 ("Dirksen"), alone or further in view of U.S. Patent Application Publication No. 2003/0139116 ("Moeggenborg 116"). We also understand that independent claim 94 also stands rejected based on Moeggenborg 100 in view of Fang and Dirksen, alone or further in view of Moeggenborg 116 and U.S. Patent Application Publication No. 2003/0119316 ("Klein").

4. We understand that the application that matured into Moeggenborg 100 was filed on September 24, 2001 and that this is considered the date of this reference for purposes of the rejection of independent claims 77, 94, and 112.

5. We declare that we conceived of and reduced to practice the inventions recited in independent claims 77, 94, and 112 prior to September 24, 2001 as discussed below.

6. Attached as Exhibit A hereto is a 47 page document entitled "Compositions for Nobel [sic] Metal Chemical-Mechanical Planarization Processes." This document has a date that is prior to September 24, 2001. This date has been redacted as evidenced by the redaction notice in the top right hand corner of Exhibit A.

7. Exhibit A describes several chemical-mechanical compositions that were made and several tests that were performed using such compositions prior to September 24, 2001. These compositions and tests illustrate the actual reduction to practice of the inventions as recited in independent claims 77, 94, and 112 in the Application. Exhibit A, pp. 3-7, 10-12, 18, 19, 23, 31, 35, and 36. These tests were performed in the context of developing chemical-mechanical polishing compositions for polishing substrates comprising noble metals. *Id.* at 1-2. Notably, the tests described in Exhibit A are also described in the currently pending Application. *See* U.S. Patent Application Publication No. 2003/0194879, paras. [0024], [0038], [0046], [0073], [0077], [0078], [0079], [0080]-[0082], [0124]-[0127], [0174], [0175], [0199], [0206], [0207], [0211], [218], and [219].

8. For example, independent claim 77 as currently pending reads as follows:

77. A combination of a chemical mechanical polishing composition in contact with a substrate surface having at least one feature thereon comprising a noble metal, said combination comprising:

a substrate comprising submicron integrated circuits and having a surface having at least one feature thereon comprising a noble metal;

said substrate surface contacting a chemical mechanical polishing composition comprising: periodic acid and an abrasive in a combined

amount sufficient to render the substrate surface substantially planar and to maintain a polishing rate between 300 Angstroms per minute to about 2000 angstroms per minute upon chemical-mechanical polishing thereof, wherein periodic acid is in an amount from about 0.05 to about 0.3 moles/kilogram.

Exhibit A describes polishing iridium (Ir), which is a noble metal, with a polishing slurry (Composition A) having 2% by weight of alumina abrasive and 0.1 mol/kg periodic acid. Exhibit A, p. 3. The removal rates obtained using Composition A were 288, 375, and 400 angstroms/minute at various polishing conditions of down force and table speed. *Id.* at 4-5.

Exhibit A also describes the use of a polishing slurry having 2 wt% alumina abrasive and 0.1 mols/kg of periodic acid (Composition Q) to polish platinum (Pt), which is also a noble metal. *Id.* at 37-39. The platinum removal rates obtained were between 1598 angstroms/minute. *Id.* at 38.

These portions of Exhibit A clearly describe compositions and tests that were actually performed using such compositions and, thereby evidence actual reduction to practice of the invention as recited in independent claim 77. Both of the tests described above involved polishing a substrate having a noble metal with a composition having an abrasive and periodic acid. The periodic acid concentration used was within the claimed range of periodic acid, and the removal rates obtained were also within the claimed range of removal rates.

9. Independent claim 94 as currently pending reads as follows:

94. A combination of a chemical mechanical polishing composition in contact with a substrate surface having at least one feature thereon comprising a noble metal, comprising:

a substrate having a surface, wherein said surface comprises a dielectric material and has at least one feature thereon comprising a noble metal, and wherein said surface is contacting a composition comprising:

periodic acid in an amount from about 0.05 to about 0.3 moles/kilogram; and an abrasive in an amount from about 0.2 to about 6 weight percent, said composition having a pH from above pH 5 to about pH 10;

and wherein on polishing the substrate surface with the composition contacting the surface the selectivity of the composition for polishing the noble metal-containing material over polishing the dielectric material is at least 1:1.

Exhibit A describes the results of several tests made with various polishing compositions (Compositions F, G, H, I, J, and K) for polishing a substrate comprising iridium, a noble metal, and TEOS (tetraethyl orthosilicate), a dielectric. *Id.* at 15-24. Each of these compositions utilized 2.3 grams of periodic acid, which converts to approximately 0.1 mols/kg. *Id.* (As a titrant was used in each of these compositions to adjust the pH, the exact concentration of periodic acid would depend upon the amount of titrant used. The amount of titrant used, however, was not significant enough to radically change the concentration of the periodic acid, which, accordingly, would still be within the claimed range of about 0.05 to about 0.3 mols/kg.) Further, the concentration of the abrasive in each of the compositions was approximately 2-2.5g (noting that two abrasives were used and that the CR-30 abrasive contained 16 weight percent alumina). This converts to approximately 2-2.5 weight percent, again, depending upon the amount of titrant used but would still be within the claimed range of about 0.2 to about 6 weight percent. (Exhibit A also describes a composition having 6 weight percent abrasive (Composition P), which would be easily understood as an abrasive concentration that could be used in a polishing composition. *Id.* at 35.) The pH of each of these compositions ranges from 6.7-7.3, and the TEOS selectivity ratio also ranges from 1:1.3 to 3.9:1.

The above referenced portions of Exhibit A clearly describe compositions and tests that were actually performed using such compositions and, thereby evidence actual reduction to practice of the invention as recited in independent claim 94. All of these tests involved polishing a substrate comprising a noble metal substrate and a dielectric with a composition having an abrasive and periodic acid as recited in claim 94. The periodic acid and abrasive concentrations and the pH for these compositions were within the claimed ranges, and the resulting selectivity ratios clearly illustrate a ratio of at least 1:1 as recited in the claim.

10. For example, independent claim 112 currently pending reads as follows:

112. A combination of a composition in contact with a substrate surface having at least one feature thereon comprising a noble metal, comprising:

A) a composition consisting essentially of:

1) water;

2) periodic acid in an amount from about 0.05 to about 0.3

moles/kilogram;

3) an alumina abrasive in an amount from about 0.2 to about 6

weight percent;

4) optionally, a pH-adjusting agent in an amount sufficient to cause the pH of the slurry to be between about 1 to about 4 or between about 5 to about 10;

5) optionally, a suspension agent;

wherein said composition is contacting

B) a substrate surface having at least one feature thereon comprising a noble metal.

Exhibit A describes several compositions and test results that evidence reduction to practice of the invention as claimed in claim 112. First, each element recited in claim 112 was reduced to practice in the compositions and tests described above in connection with claim 94, and further including Composition E, *Id.* at 13-15, noting that (i) each composition contained water, (ii) the abrasive was alumina, (iii) ammonium hydroxide was used to adjust the pH of these compositions to 6.7-7.3, and (iv) each composition contained a suspension agent comprising various compositions (*e.g.*, Alumina-C, Darvan C, Ludox TM50, ethyl carbonate, succinic acid, and CR140), *Id.* at 13-24 (in particular noting the description regarding Composition E and the use of the second abrasive for better suspension).

In addition, Composition B comprised, 2 weight percent alumina abrasive and 0.1 mol/kg periodic acid, and the pH was adjusted with tetramethyl ammonium hydroxide to a between 6 and 7. *Id.* at 7. This composition was used to polish iridium, a noble metal. *Id.* at 7. Further, Composition D describes using a polishing slurry composition to polish iridium. *Id.* at 10-12. This composition comprises 2 weight percent alumina abrasive and 0.1 mols/kg periodic acid. *Id.* at 12. The pH of this slurry is adjusted with ammonium hydroxide to a pH of about 3-4. *Id.*

Further, Composition N describes a platinum (noble metal) polishing composition comprising 2 weight percent alumina abrasive and 0.1 moles/kg of periodic acid at a pH of 1.6. *Id.* at 31.


These portions of Exhibit A clearly describe various compositions and test that were actually performed using such compositions and, thereby evidence actual reduction to practice of the invention as recited in independent claim 112. These tests involved polishing a substrate having a noble metal with a composition having an alumina abrasive and periodic acid as recited in claim 112. The abrasive and periodic acid concentrations were within the ranges recited in claim 112. The pH of the various slurry compositions were either between about 1 to about 4 or between about 5 to about 10 as recited in claim 112. Further, these compositions utilized various suspension agents as recited in claim 112.

11. We declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated: _____

Robert J. Small

Dated: Jun. 27, 2007



Zheifei J Chen